

**Synthesis, Microstructure and Properties
of Nickel Aluminide Foams**

Summary of Research for NASA grant NCC3-870

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1. Executive Summary

- Two Ph.D. students were involved in the project: Mr. Christopher Schuh (part-time, graduated in Spring 2001) and Ms. Andrea Hodge (full-time, graduated Summer 2002). One post-doctoral fellow, Dr. Heeman Choe, worked full-time on the project from July to December 2002.
 - A new process to aluminize and chromize nickel foams was created.
 - A kinetic aluminization model was developed.
 - Creep testing was conducted on the foams.
 - A finite-element model and a simplified analytical model for foam creep were produced.
 - Four articles were written: one is published, two are accepted for publication, and one is in preparation.
 - Ms. Hodge spent four months at NASA Glenn Research Center (9-12/2001 and 2-3/2002) under the supervision of Dr. Nathal. She conducted research on NiAl foam fabrication, mechanical testing and numerical modeling. She gave a talk at the ASM annual conference in November 2001 and presented her results at NASA in December 2001.
1. D.C. Dunand, A.M. Hodge, C. Schuh, "Pack Aluminisation Kinetics of Nickel Rods and Foams", *Materials Science and Technology*, **18**, 326-332 (2002).
 2. Hodge, D. C. Dunand "Measurement and Modeling of Creep in Open-Cell NiAl Foams", *Metallurgical and Materials Transactions*, accepted for publication.
 3. H. Choe, D.C. Dunand, "Superalloy Foams Produced by Gas-Phase Alloying Technique", *MetFoam 03 - 3rd International Conference on Cellular Metals and Metal Foaming Technology*, accepted for publication.
 4. H. Choe, D.C. Dunand, "Processing, Structure and Mechanical Properties of Superalloy Foams", in preparation.

2. Foam Processing

Two grades of commercially-available nickel foams were used (Table 1). These foams have open-cell with hollow struts (Figure 1a), so the diffusion distance is given by the strut wall thickness and is small. Small samples of near-stoichiometric NiAl foams (Ni-31wt.%Al) were produced by pack-aluminization of these foams (Figure 1b) and EDS analysis was performed to measure layer growth and composition for kinetic studies. A kinetic model was established and predictions compared to experimental data (Figure 2).

The aluminum range for nickel-base superalloys is ca. 4-9 wt.%Al. Ni-8wt.% Al foams were produced by pack aluminization and, after homogenization and precipitation heat-treatments, exhibited the γ/γ' structure typical of superalloys. Additional pack-chromizing resulted in Ni-15wt.%Cr-8wt.%Al foams, also with the γ/γ' structure. Details of the process are given below.

Table 1. Geometric Parameters of Nickel Foams Used for Aluminization

Pores per Linear Inch (ppi)*	Foam Thickness (mm)	Cell Diameter (mm)**	Strut Width (μm)	Strut Wall Thickness (μm)	Relative Density (%)	Supplier
30	25.4	0.85	117 \pm 21.4	40.5 \pm 6.5	2.8	Astro Met
20	25.4	1.27	224 \pm 34	83.5 \pm 21.3	2.2	"

* Data from supplier

** Calculated from ppi value

Using the aluminization method developed for the thin specimens used in the above microstructure study proved to be inadequate for the thicker mechanical testing specimens (10x10x22 mm), due to uneven deposition of Al at different depths within the Ni foam. A new pack composition was determined: 3 wt% NH_4Cl , 15 wt.% Al-Ni (50 wt. %) powder, 82 wt.% Al_2O_3 powder (the main difference is the replacement of pure Al with alloyed Al-Ni powders). The pack and nickel foams were placed in a stainless steel bag, which was introduced in a furnace under flowing argon. A first aluminization treatment lasted for 4 hours for the 30 ppi foam and 1.5 hours for the 20 ppi foam, both at 1034°C. An additional aluminization treatment followed, corresponding to 4 hours of aluminization and additional annealing time where no aluminum is added to the foam, but the Al composition gradient is eliminated by diffusion (homogenization). The total time at 1034°C for the 20 and 30 ppi foams was 40 and 20 hours, respectively. The resulting microstructure is single-phase NiAl throughout the foam (Figure 3a). For superalloy foams, the same pack was used for aluminization for shorter times, and another pack was used for chromization: 5 wt% NH_4Cl , 25 wt.% Cr powder, 70 wt.% Al_2O_3 powder. The temperature used was 1000°C, and times ranged from 1 to 3.5 h. (resulting in foam compositions of 13-20 wt.% Cr). To fully homogenize the foams, a heat-treatment followed at 1200°C for 120 h., terminated by brine quenching. The resulting supersaturated foam was aged at ca. 900 °C for ca. 7.5 h. (depending on exact composition), resulting in precipitation of γ' precipitates (Figure 3b).

3. Compression Creep Test

Compression creep test were conducted at Northwestern University and NASA Glenn Research Center, using both 20 and 30 ppi NiAl foams, with length to width ratio of 2. Tests were conducted in air from 827 to 1100°C for stresses ranging from 0.1 to 1.5 MPa. A stress exponent of 3-4 and an activation energy of $Q=195 \pm 19$ kJ/mol was measured, similar to values reported for bulk NiAl (Fig. 4a)

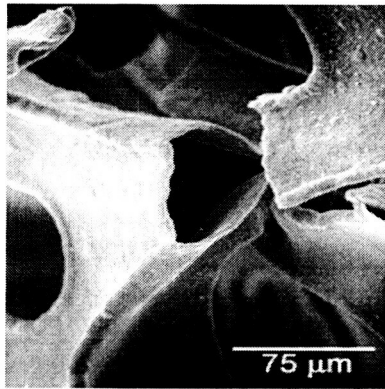
For Ni-Al superalloy foams, creep tests were carried out in air at Northwestern University for various temperatures (680 to 825°C) and stresses (~ 0.1 to ~ 0.3 MPa), resulting in a stress exponent of 5.3 and an activation energy of 260 kJ/mol, again in agreement with bulk values. For Ni-Cr-Al foams, the stress exponent is somewhat lower, but the activation energy is the same (Fig. 4b).

4. Modeling

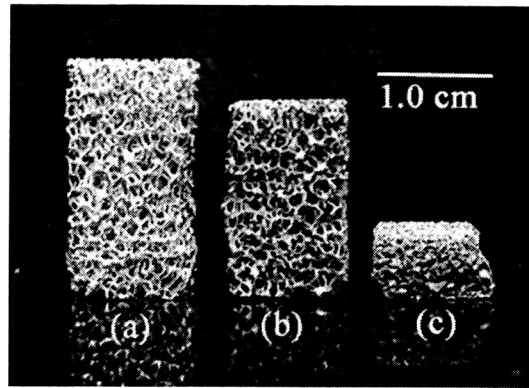
A simple analytical model was developed, assuming that the applied load is transmitted through vertical struts, which span the whole height of the sample: these struts deform by uniaxial compression while the horizontal struts do not deform. This model gives surprisingly good results when compared to data (factor of two error), and is much more accurate than another model by Gibson and Ashby (factor of hundred error). The latter model assumes that the applied load transmitted through vertical strut results in the bending of the corresponding horizontal struts.

A 3-dimensional finite-element model (FEM) was used to simulate the creep behavior of a NiAl foam consisting of cubic cells with solid or hollow struts (Fig. 5a). The FEM creep rate predictions are slower by a factor of two as compared to experimental data (Fig. 5b). Predictions are sensitive to foam density and strut geometry (hollow vs. solid). The analytical model described above produces results that are very close to the predictions of the FEM numerical model and in good agreement with the experimental data.

5. Figures



(a)



(b)

Fig. 1a. SEM micrograph showing hollow strut in as-received Ni foam.

Fig. 1b. NiAl foam (a) as processed and (b,c) deformed at 900 °C.

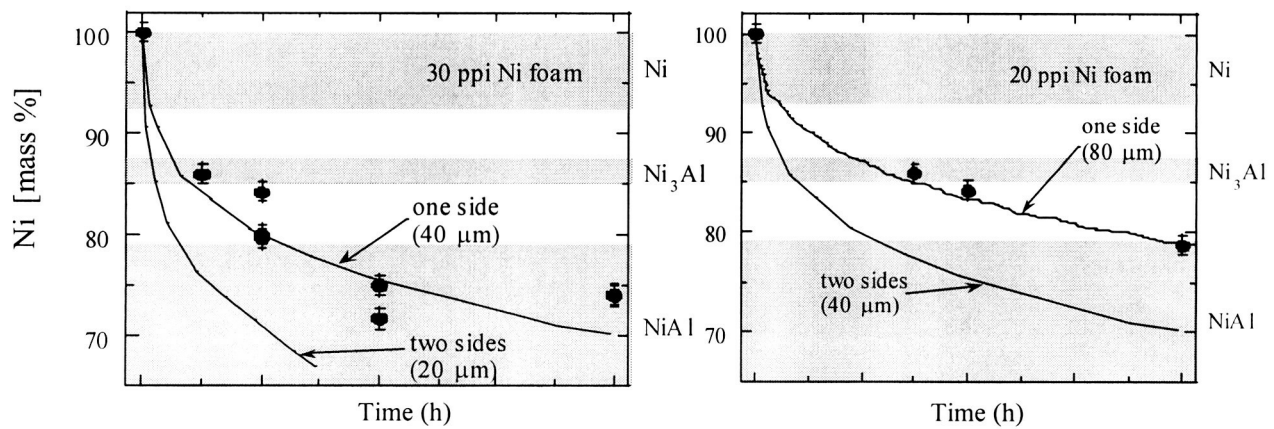
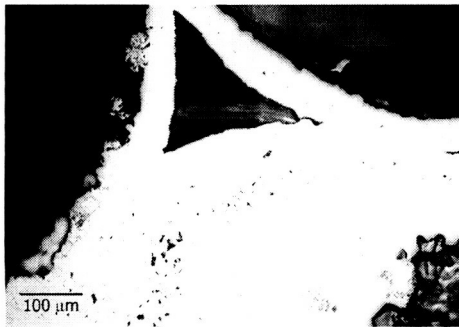
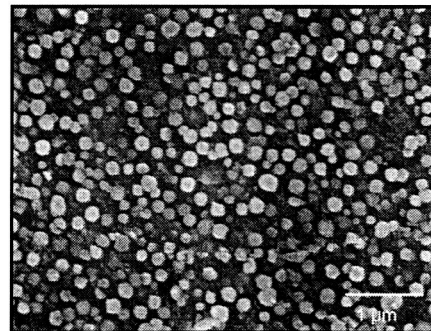


Fig. 2. Foam composition as a function of aluminization time at 1000 °C: experimental data are in good agreement with kinetic model assuming diffusion from one side of the foam struts.



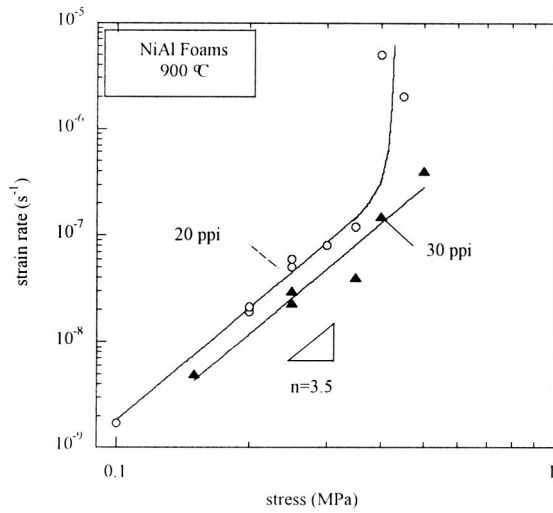
(a)



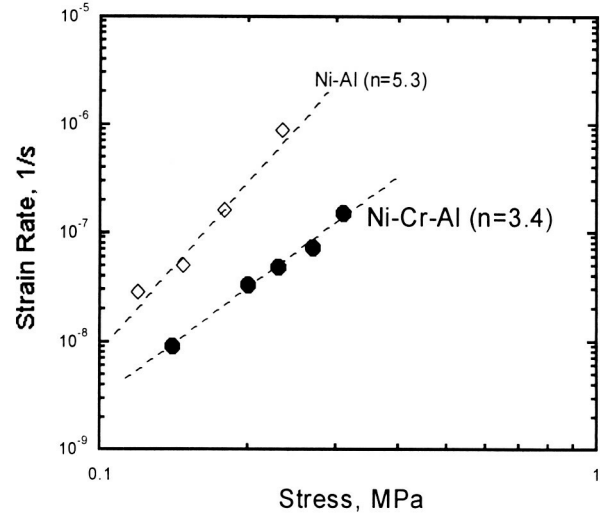
(b)

Fig. 3a: Cross-section of strut in NiAl foam after homogenization, showing single-phase NiAl structure.

Fig. 3b: Microstructure of strut in Ni-18wt.%Cr-5.3wt.%Al after heat-treatment, showing γ/γ' superalloy structure.



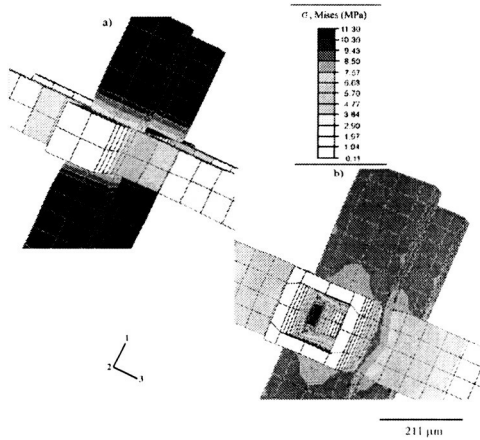
(a)



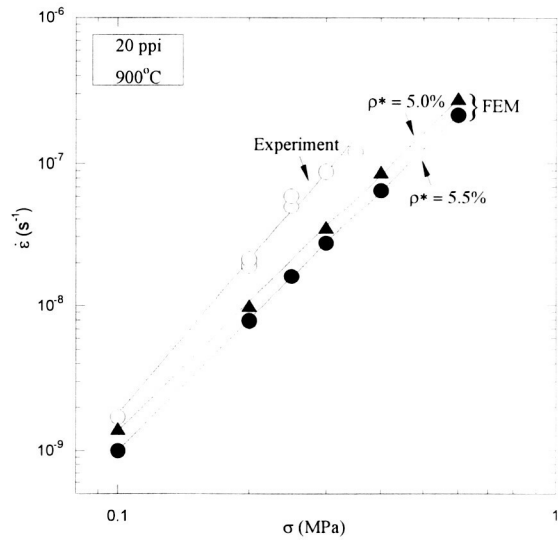
(b)

Fig. 4a: Stress vs. minimum strain rate at 900 °C for 20 and 30 ppi NiAl foams (power-law breakdown occurs for 20 ppi foams at high stress).

Fig. 4b: Stress vs. minimum strain rate at 825 °C for superalloy foams.



(a)



(b)

Fig. 5a: FEM model showing Von Mises effective stress for an external stress $\sigma^* = 0.2$ MPa at 900 °C after achieving minimum strain rate for foams ($\rho^* = 5.5$) with solid and hollow struts.

Fig. 5b - Compressive strain rate vs. stress curves at 900 °C as measured experimentally on 20 ppi foams (with relative density $\rho^* = 5.0-5.5\%$) and as calculated by FEM ($\rho^* = 5.0$ and 5.5%) for hollow struts.